

Table 6. XRD* sampling and analytical methods for crystalline silica

Item	NIOSH Method 7500	OSHA Method ID-142	MSHA Method P-2	MDHS 51/2
Silica polymorph	Quartz, cristobalite, tridymite	Quartz, cristobalite	Quartz, cristobalite	Quartz
Sampler	10-mm nylon cyclone, 1.7 L/min; Higgins-Dewell cyclone, 2.2 L/min	10-mm nylon Dorr-Oliver cyclone, 1.7 L/min	10-mm nylon Dorr-Oliver cyclone, 1.7 L/min	Higgins-Dewell cyclone, 1.9 L/min
Filter	37-mm, 5- μ m PVC membrane	37-mm, 5- μ m PVC membrane	37-mm, 5- μ m PVC membrane	25-mm, 5- μ m PVC membrane
Volume	400–1,000 L; total dust < 2 mg	408–816 L; total dust < 3 mg	400–1,000 L; total dust < 3 mg	\geq 456 L; total dust < 2 mg
Filter preparation	RF plasma asher, muffle furnace, or filter dissolution in THF	Dissolve filter in THF	RF plasma asher	None
Redeposition	On 0.45- μ m silver membrane filter	On 0.45- μ m silver membrane filter	On 0.45- μ m silver membrane filter	None
Drift correction	Silver internal standard	Silver internal standard	Silver internal standard	External standard (e.g., aluminum plate)
X-ray source	Cu K $_{\alpha}$; 40 kV, 35 mA	Cu K $_{\alpha}$; 40 kV, 40 mA	Cu K $_{\alpha}$; 55 kV, 40 mA	Cu K $_{\alpha}$; 45 kV, 45 mA
Calibration	Suspensions of SiO $_2$ in 2-propanol (deposited on silver membrane filter)	Suspensions of SiO $_2$ in 2-propanol (deposited on silver membrane filter)	Suspensions of SiO $_2$ in 2-propanol (deposited on silver membrane filter)	Sampling from a generated atmosphere of standard quartz dust
Proficiency testing	PAT	PAT	PAT	WASP
Range (μ g quartz)	20–2000	50–160 (validation range)	20–500	50–2000
LOD (μ g quartz)	5 (estimated)	10	5	3
Precision	$\overline{\text{RSD}} = 0.08$ 50–200 μ g	$\overline{\text{CV}} = 0.106$ @ 50–160 μ g	$\text{CV} = 10\%$ @ 20–500 μ g	$\text{CV} = 5\%$ @ 50 μ g

*Abbreviations: Cu = copper; CV = coefficient of variation (equivalent to RSD); $\overline{\text{CV}}$ = pooled coefficient of variation; K $_{\alpha}$ = electron ionization energy; kV = kilovolt(s); LOD = limit of detection; mA = milliampere(s); MDHS = Methods for the Determination of Hazardous Substances (Health and Safety Executive, United Kingdom); MSHA = Mine Safety and Health Administration; NIOSH = National Institute for Occupational Safety and Health; OSHA = Occupational Safety and Health Administration; PAT = proficiency analytical testing; PVC = polyvinyl chloride; RF = radio frequency; RSD = relative standard deviation; $\overline{\text{RSD}}$ = pooled relative standard deviation (equivalent to $\overline{\text{CV}}$); THF = tetrahydrofuran; WASP = Workplace Analysis Scheme for Proficiency; XRD = X-ray diffraction.

Table 7. IR* sampling and analytical methods for crystalline silica

Item	NIOSH Method 7602	NIOSH Method 7603	MSHA P-7	MDHS 37	MDHS 38
Matrix		Coal mine dust	Coal mine dust		
Sampler	10-mm nylon cyclone, 1.7 L/min; Higgins-Dewell cyclone, 2.2 L/min	10-mm nylon cyclone, 1.7 L/min; Higgins-Dewell cyclone, 2.2 L/min	10-mm nylon Dorr-Oliver cyclone, 2.0 L/min	Higgins-Dewell cyclone, 1.9 L/min	Higgins-Dewell cyclone, 1.9 L/min
Filter	37-mm filter; 5-µm PVC or MCE membrane	37-mm filter; 5-µm PVC membrane	37-mm filter; 5-µm PVC membrane, preweighed	37-mm filter; 5-µm PVC membrane	37-mm filter; 5-µm PVC membrane
Volume	400–800 L; total dust <2 mg	300–1,000 L; total dust <2 mg	Not stated	≥456 L; total dust <1 mg	≥456 L; total dust <0.7mg
Filter preparation	RF plasma asher or muffle furnace	RF plasma asher or muffle furnace	RF plasma asher	None	Muffle furnace
Analytical sample preparation	Mix residue with KBr, press 13-mm pellet	Redeposit on 0.45-µm acrylic copolymer filter	Redeposit on 0.45-µm acrylic copolymer filter	None	Mix residue with KBr, press 13-mm pellet
Standard	Polystyrene film	Polystyrene film	Polystyrene film	Polystyrene film	Polystyrene film
Calibration	Quartz diluted in KBr	Standard suspension of quartz in 2-propanol	Standard suspension of quartz in 2-propanol	Sampling from a generated atmosphere of standard quartz dust	Sampling from a generated atmosphere of standard quartz dust
Proficiency testing	PAT	PAT	PAT	WASP	WASP
Range (µg quartz)	10–160	30–250	25–250	10–1,000	5–700
LOD (µg quartz)	5 (estimated)	10 (estimated)	10	Varies with particle size	Varies with particle size

See footnote at end of table.

(Continued)

Table 7 (Continued). IR* sampling and analytical methods for crystalline silica

Item	NIOSH Method 7602	NIOSH Method 7603	MSHA P-7	MDHS 37	MDHS 38
Precision	<u>RSD</u> <0.15 @ 30µg	<u>RSD</u> = 0.098 @ 100–500µg	CV = 5–10 % @ 100–500 µg	CV = 5 % @ 50 µg	CV = 5 % @ 50 µg

*Abbreviations: CV = coefficient of variation (equivalent to RSD, relative standard deviation); IR = infrared absorption; KBr = potassium bromide; MCE = methyl cellulose ester; MDHS = Methods for the Determination of Hazardous Substances (Health and Safety Executive, United Kingdom); MSHA = Mine Safety and Health Administration; NIOSH = National Institute for Occupational Safety and Health; LOD = limit of detection; PAT = proficiency analytical testing; PVC = polyvinyl chloride; RF = radio frequency; RSD = pooled relative standard deviation (equivalent to CV, pooled coefficient of variation); WASP = Workplace Analysis Scheme for Proficiency.

Table 8. Intralaboratory results for evaluation of XRD silica method

Item	Filter loading		
	69.4 µg	98.4 µg	204 µg
Degrees of freedom	12	11	12
RSD for sampling and analytical methods (%) ^{*,†}	8.8	6.3	8.1

Source: NIOSH, BOM [1983].

^{*}RSD = relative standard deviation. RSD for sampling and analytical methods represents the RSD in mass estimates, accounting for intersampler and analytical variability.

[†]Implications for XRD: Pooled filter levels and pump error (assumed to be <5%) indicate that the overall imprecision is as follows: Total RSD for sampling and analytical methods is 9.3%. Therefore, the upper 95% confidence limit on the accuracy (35 degrees of freedom) is 21%.

Table 9. Intralaboratory results for evaluation of IR silica method

Item	Filter loading		
	67.2 µg	99.7µg	161 µg
Degrees of freedom	10	12	11
RSD for sampling and analytical methods (%) ^{*,†}	5.8	7.8	7.4

Source: NIOSH, BOM [1983].

^{*}RSD = relative standard deviation. RSD for sampling and analytical methods represents the RSD in mass estimates, accounting for intersampler and analytical variability.

[†]Implications for IR: Pooled filter levels and pump error (assumed to be <5%) indicate that the overall imprecision is as follows: Total RSD for sampling and analytical methods is 7.1%. Therefore, the upper 95% confidence limit on the accuracy (33 degrees of freedom) is 17%.

Table 10. XRD method evaluation: concentration ranges bracketing applicable exposure limits for which the NIOSH accuracy criterion is met*
($\mu\text{g}/\text{m}^3$)

Cyclone and sampling rate	Filter loading			Applicable exposure limit
	69.4 μg	98.4 μg	204 μg	
Nylon cyclone, 1.7 L/min	85	121	251	100
GK2.69 cyclone, 4.2 L/min	34	49	102	50

*Eight-hour sampled masses are combined with results of NIOSH, BOM [1983].